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Faculty of Textile Engineering

# SELECTED MECHANICAL PROPERTIES OF POLYMERIC OPTICAL FIBER (POF)

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SUMMARY OF THE THESIS

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| **Field of study:** | Materials Engineering |
| **Mode of study:** | Full time |
| **Department :** | Department of Material Engineering |
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# Abstract

The integration of polymeric optical fiber (POF) into fabrics has brought a lot of interest in textile design. POF fabrics could be applied to display the profiles of human beings, animals, objects (warning devices), obstacles (steps and carpets) and the like in places with poor visibility. However, this integration is facing huge problems. The major problems are derived from the poor flexibility, drapability, durability and side illumination of POF fabrics. The Properties of POF are considered as the critical factors which would influence the manufacturing processes and properties of POF fabrics. Compared with traditional textile yarns or filaments, POF is relatively brittle, stiff, and sensitive to bend due to its thick diameter. At present, the diameter of end emitting POF in weaves, knits and embroideries is generally in the range of 0.2 ~ 1.0 mm, the diameter of side emitting POF applied in safety applications (corridors and obstacles) varies in the range of 2 ~ 6 mm or above. The big challenge is to manufacture POF with sufficient flexibility and good side illumination intensity. The side illumination intensity of POF is usually enhanced by surface modifications (chemically and mechanically) or by using the fluorescent fabric cover which could also improve the resistances of naked POF to external environment such as mechanical damage and UV radiation.

This thesis work is aimed to investigation the selected mechanical properties of POF based on the less contributions from the standpoint of the properties of POF integrated fabrics at present, rather than propose new methods to improve the side illumination of POF or propose new manufacturing techniques of POF fabrics. The experimental work starts from tensile testing and the results indicate that, there is an inverse relation between fiber diameter and tensile strength of POF. The strain value decreases as the fiber diameter increases. And the modulus varies significantly and is assumed to be determined by the various changes of tensile strength and strain. As a synthetic polymer fiber, however, POF is not uniform in fiber thickness, the results from strength distribution represent that the gauge length plays an important role in tensile strength. The results evaluated by Weibull distribution indicate that there is a decay exponential relation between tensile strength and gauge length. POF is with the core/cladding structure. The contributions of core and cladding to the mechanical properties of the whole fiber, and the interphase property between core and cladding are investigated by nanoindentation technique. It is observed from the experimental data that the core is harder than the cladding. Both core and cladding show very strong loading rate sensitivity during nanoindentation testing, which could be explained by the visco-elastic properties of polymers. The interphase width is estimated to be in the range of 800 ~ 1600 nm roughly. In the investigation of POF durability, two fatigue testing are taken into account. One is the tension fatigue testing which is applied to measure the strain response of POF under constant stress amplitude. The results demonstrate that both cyclic extension and total extension go up with increasing fatigue cycles. Compared with other fibers, while, 0.5 mm POF has higher total extension but lower cyclic extension than thicker POFs, which could be explained by different applied external stress and different amount of irreversible deformation in each fiber during fatigue testing. Another is the flex fatigue testing, which is aimed to investigate the flex fatigue lifetime based on the number of bending cycles to break by using the model of fatigue life curve. It is estimated that the fatigue lifetime could be influenced significantly by the testing condition such as the bending angle and speed. In the meanwhile, the flex fatigue sensitivity coefficient is also evaluated and compared with the general value for other materials.

**Keywords:**

polymeric optical fiber; strength distribution; nanoindentation properties; tension fatigue; flex fatigue

# Anotace

Integrace polymerních optických vláken (POF) do textilií je přínosem z hlediska designu na jedné straně, ale na druhé straně umožňuje také zviditelnění obrysů osob, zvířat, předmětů, vymezení překážek (schody, kraje koberců) apod. Při zabudování optických vláken do textilií se sleduje zejména jejich ohebnost, trvanlivost a intenzita vyzařování. Ve srovnání se standardními textilními materiály (příze, hedvábí) jsou některá POF relativně křehká, tuhá a citlivá na ohyb v závislosti na jejich průměru. V současné době je průměr běžně využívaných POF pro tkaniny, pleteniny a výšivky v rozmezí od 0,2 do 1,0 mm. Pro integraci do oděvních textilií s cílem zviditelnění osob lze použít stranově vyzařující optická vlákna o průměru 2-6 mm a např. Pro osvětlení chodeb a vymezení překážek je možno použít optická vlákna o průměru od 6 mm výše. Pro uvedené aplikace je nutno vždy hledat kompromis mezi dostatečnou ohebností a světelným výkonem vláken. Pro zvýšení intenzity vyzařování se používá pokrytí povrchu stranově vyzařujících vláken textilním potahem. Vlákna jsou umístěna v dutině tkaniny nebo opletena textilními přízemi. Textilní potah současně chrání optické vlákno před mechanickým poškozením a vlivem UV záření.

Disertační práce je zaměřena na zkoumání vybraných mechanických vlastností POF. Experimentální práce je založena nejprve na zkoumání tahových vlastností stranově vyzařujících optických vláken v závislosti na jejich průměru. S rostoucím průměrem vlákna se relativní pevnost a tažnost snižuje. Modul optických vláken se mění významně spolu se změnami pevnosti a tažnosti. Podobně jako u syntetických polymerních vláken ovlivňuje také upínací délka pevnost polymerních optických vláken. Výsledky získané na základě Weibullova rozdělení indikují exponenciální pokles pevnosti v závislosti na upínací délce. POF mají strukturu jádro/plášť. Příspěvek této struktury i vlastností na rozhraní povrchů mezi jádrem a pláštěm k mechanickým vlastnostem POF byl zkoumán s využitím nanoindentační metody. Bylo zjištěno, že jádro POF je tvrdší než plášť. Obě komponenty, jak jádro, tak i plášť indikují velmi silnou citlivost na rychlosti zatěžování v průběhu nanoindentačního testu, která může být popsána pomocí visko-elastických vlastností polymerů. Odhad šířky mezifáze je přibližně v rozmezí od 800 ~ 1600 nm. Při hodnocení ohebnosti a životnosti (únavy) POF, byly vzaty v úvahu dva typy testování. Nejprve bylo testováno cyklické namáhání založené na měření deformační odezvy POF na konstantní amplitudu zatěžování. Výsledky ukazují, že jak cyklické protažení, tak i celkové protažení souvisí s přírůstkem únavových cyklů. Ve srovnání s jinými vlákny vykazuje POF o průměru 0,5 mm vyšší celkové protažení, ale nižší cyklické protažení, než POF s větším průměrem. To by mohlo být vysvětleno různým množstvím nevratné deformace každého vlákna v průběhu testování únavy. Dále bylo provedeno testování odolnosti v ohybu dle počtu ohybových cyklů do přetrhu. Bylo ukázáno, že tato veličina je významně ovlivněna podmínkami testování, což je úhel ohybu a rychlost. Byl hodnocen také koeficient ohybové citlivosti a porovnán s hodnotami běžnými pro jiné materiály.

**Klíčová slova:**

*polymerní optické vlákno, rozložení pevnosti, nanoindentační vlastnosti, únava při cyklickém namáhání v tahu, únava při opakovaném namáhání v ohybu*

# Summary

# 1 Introduction

Polymer/plastic optical fiber (POF), made of polymers or plastics, was firstly introduced in the 1960s as a substitution of glass optical fiber in data communication in a short distance generally less than 1 km. POF was not utilized universally due to its high optical attenuation. However, POF has received enough attention in the 1990s because of the development of graded-index POF and the achievement of low attenuation [1-4], combined with the successive improvements in both transparency and bandwidth, POF is recently applied as a high-capacity transmission medium [5]. At present, the applications of POF have increased significantly. Apart from the application in data transmission, POF is widely used in optical components (such as optical switches, amplifiers and tunable optical sources), and other extended fields such as therapy and textile.

Textiles can be classified into three categories based on the end uses: clothing textiles, decorative textiles and technical textiles. The demand for textiles has increased dramatically during the last two decades due to the rise in living standard of human beings. However, the increasing demand has brought a big challenge to develop new materials or introduce existed materials to textiles. Even though glass fiber based textile materials have been known for quite a long period of time, the idea of optical fiber based fabrics was arose at the end of twentieth century. The initial optical fiber based fabrics were manufactured for end illumination by cutting the optical fiber at the required point of light emission. Visually, the optical effect on POF based textile fabrics was purely aesthetic. The color, brilliance or shine of POF fabric could be changed from the light reflection on fabric surface with different fiber materials, fabric pattern and fabric density [6]. Recently, following with the development of POF itself and the manufacturing techniques of POF fabric, POF integrated textiles have extended the applications from the photo-metric fields for illumination to the radiometric fields for sensing [7].

At present, there are two major applications of POF in textile fabrics. One is utilized as an active lighting element in fabric structure for lighting purpose, the application fields include indoor [8] and outdoor lighting [9], safety [10], fashion and design [11], displays [12] and medical technology [13]. Another is used as an optical sensor in fabric structure for sensing purpose. Selected applications regarding these areas are introduced as follows. Generally, the textile integrated POF sensors are aimed at measuring the physical responses such as pressure [14], stress [15] and strain [16], or applied for biomedical responses based on biological parameters such as breathing [17], sweat [18] and oxygen content [19].

There are numerous advantages of integrating POFs into traditional fabric structures. First of all, POFs make the fabrics luminous. POF fabrics could emit light not only on the fabric surface but also at required points based on the macrobends of POF or additional surface modifications. In contrast to general electrical products, POF fabrics are immune to electromagnetic interference (EMI), free of electricity and heat. At the same time, POF fabrics can still keep the textile appearance. The dimension of luminous area is flexible, which could be small in centimeters for embroideries or large in meters for weaves and weft knits. Additionally, the separation of light source and POF medium generates simple connection and easy handling of POF fabrics. Furthermore, the use of POFs instead of glass optical fibers in luminous fabrics is beneficial to the flexibility, light weight, durability and small injuries [20].

On the other hand, POF fabrics have some disadvantages. Even though POF fabrics are popular in illumination, decoration, radiation and sensing applications, a lot of potential applications are highly restricted due to the limitations of POF itself. The bendability of POFs is not sufficient enough as traditional yarns, which limits a lot of possibilities in structure design. Thin POFs with side illuminating effect are not commercially available on the market due to the complicated manufacturing processing and poor transmission rate of light rays. In addition, the mechanical properties of POFs are not satisfied at sub-zero temperature. The thermal stability of POFs is problematic that limits the working temperature significantly. Furthermore, it is still a challenge to reduce the optical loss of POFs.

# 2 Purpose and the current state of the problem

As a synthetic polymeric fiber, POF is expected to be uniform in thickness. As a matter of fact, the fiber diameter, the cladding thickness, as well as the surface roughness are not the same along the fiber length due to the manufacturing processes, packing processes and so on. These variations are difficult to control and could have unexpected influences on the mechanical properties of POF. There are five aspects selected in mechanical properties to study the effects of fiber diameter and the durability of POF: tensile properties, strength distribution, local mechanical properties of both core and cladding and the interphase property between them, tension fatigue properties and flex fatigue behaviors. The goals of this work are to survey the selected mechanical properties of POF which are referred in the applications of POF fabrics and discussed from the point of view of textile background, rather than offer detailed and standard methodologies to investigate the mechanical properties of POF, or provide new methods of improvement of POF attenuation, or propose new technologies to manufacture POF fabrics. It is aimed to introduce POF to textile fields mainly, present basic and important knowledge of POF itself regarding mechanical properties, give information to POF manufacturers, and provide links to future for better research work and boarder applications in textiles.

### *Tensile properties*

To investigate the effects of fiber diameter on tensile testing of POF. To understand the mechanism of tensile failure of POF.

### *Strength distribution*

To investigate the effects of fiber length on tensile strength of POF. To estimate the relationship between fiber length and tensile strength by Weibull distribution.

### *Local mechanical properties*

To figure out the contributions of each part (core or cladding) to the properties of the whole fiber by nanoindentation technique. To discuss the creep deformation of POF due to the inherent visco-elasticity of polymer materials. To study the interphase properties between core and cladding.

### *Tension fatigue*

To estimate the strain response under constant load amplitude. To investigate the tensile properties after tension fatigue testing of POF without fiber fracture.

### *Flex fatigue*

To explore a new method to investigate the flex fatigue of POF. To evaluate the flex fatigue properties of POF by fatigue life model. To study the flex fatigue sensitivity coefficient of POF based on an empirical equation.

# 3 Overview of the current state of the problem

As mentioned above, there are a great deal of applications of POFs in textiles. In the field of POF fabrics, a lot of potential has been restricted by the properties of POF, which not only influence the illumination properties of POF fabric, but also limit the possibilities of integration of POF into fabrics. For example, it is still problematic to commercially manufacture side emitting POFs with diameter less than 0.2 mm. Even though POFs with diameter more than 1 mm could be used as active illuminating elements in emergency or safety textiles in order to give enough light rays in special dark places [10]. The possibility to apply POFs into traditional fabric structures is obviously lower with thicker POFs. Moreover, the bendability of POFs, the technique processing of POF fabric, the illuminating effect, the drapability of POF fabric are influenced by POF properties more or less.

In practical illumination and decoration applications of POF fabrics, the POF diameter used as traditional textile yarns or fibers normally varies from 0.2 mm to 1 mm. In order to obtain clear luminous patterns, the illuminating effect is generally achieved by the macrobends or additional treatments of POFs in woven, weft-knitted and embroidered fabric structures. Generally speaking, in weaves, POFs are laid straightly, the light illumination is obtained by surface modifications and the light loss is quite low; in weft knits (knitted webs/meshes), POFs are arranged in bending shapes, the light illumination is obtained by macrobends and the light loss is higher compared to the first case; while in embroideries, POFs are either bent or set in any free form, the light illumination is achieved by macrobends of POFs and the light loss is highest in all cases. Both mechanical properties and light loss restrict the dimension and market prospects of POF fabrics.

A lot of contributions have been devoted to the manufacturing technology of POF fabrics, the enhancement of side illumination of POFs or POF fabrics, and the improvement of optical loss of POFs induced by mechanical deformations (tensile, bend or compression) of POF. It seems that how to develop the POF fabrics and how to obtain high intensity lateral light on POF fabrics have been catching more attention. However, how the POF properties influence the development and properties of POF fabrics is also very interesting and vital. There are very less literatures focusing on the mechanical properties of POF with a core/cladding structure, the flexibility and the durability of POF itself in details so far, which are important and unresolved issues required to be explored urgently.

# 4 Methods used, studied material

## 4.1 Materials

The materials employed in this thesis were naked POFs, prepared by Grace POF Co., Ltd., Taiwan. Table 4.1 shows the basic characterization of POFs. All POFs display the same structure that possess core and cladding layers. All cores have the same polymer that is PMMA, all claddings have the same composition which is blended by PMMA and polytetrafluoroethylene (PTFE/Teflon), and the corresponding refractive indices are 1.49 and 1.42, respectively. There are five diameters in total, the minimal bending radii of all POFs are the same and eight times of fiber diameter.

Table 4.1 Technical data of all POFs.

| Basic properties | Grace POF |
|---|---|
| Core material | PMMA |
| Cladding material | PMMA/Teflon |
| Jacket material | no |
| Fiber diameter [mm] | 0.25/0.4/0.5/0.75/1.0 |
| Core refraction index | 1.49 |
| Cladding refraction index | 1.42 |
| Numerical aperture | 0.44 |
| Wavelength [nm] | 400 ~ 780 |
| Limit of bending radius | 8 × fiber diameter |

## 4.2 Methods

***Tensile testing***. The basic mechanical properties of single fiber regarding the tensile properties were investigated at first. The stress-strain experiments for all POFs were carried out on Instron at 20 ˚C and 65% relative humidity. The testing speed was set as 300 mm/min. The gauge length was 100 mm. 50 times were averaged for each.

***Strength distribution***. The relationship of fiber strength and gauge length of 0.75 mm POF was investigated by Instron at 20 ˚C and 65% relative humidity. The testing speed was 100 mm/min. The gauge lengths were chosen as 30, 50, 75, 100, 150 and 200 mm. 50 times were averaged for each.

***Nanoindentation testing***. The nanoindentation testing in terms of hardness property, creep deformation and interphase property between core and cladding of 0.5 mm POF were proceeded by Hysitron with a three-side pyramidal Berkovich diamond indenter. The typical load-displacement curve of POF is presented in Figure 4.1. The effects of fiber diameter and cross section direction on hardness property were also discussed.

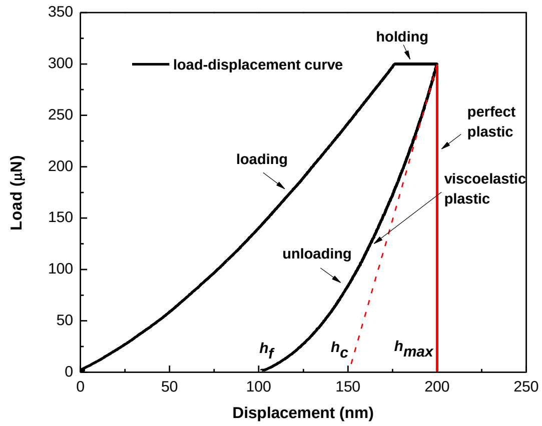


Figure 4.1 Load-displacement curve ($h_{max}$-maximum depth, $h_c$-contact depth, $h_r$-final depth).

The nanoindentation testing for 0.5 mm POF was conducted in two ways: when the holding time $t_H$ was 10 s, the loading time $t_L$ varied from 5 s to 30 s; when the loading time was 10 s, the holding time shifted from 5 s to 30 s. For both ways, the unloading time was the same as the loading time and the maximum load was set as 0.3 mN.

The interphase properties between core and cladding in POF was also investigated by nanoindentation technique. The maximum nanoindentation depths were 120, 80, 40 nm and relevant spacings of 1900, 1300, 700 were used to avoid overlapping of plastic deformation zones between adjacent indents. POFs were tested from cladding to core in the line through the centre of cross section.

***Tension fatigue testing***. In this investigation, the tension fatigue testing of selected POFs was proceeded by Instron at 20 ˚C and 65% relative humidity. Each sample was measured with constant applied load related to its ultimate tensile strength. The loading time was the same as unloading time, which was 2.5 s. The initial gauge length was 100 mm. 20 times were averaged for each.

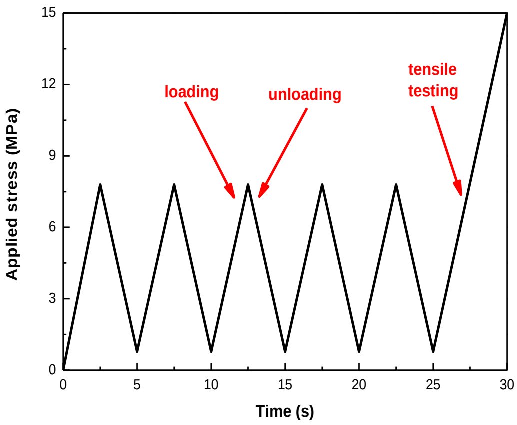


Figure 4.2 Testing design for tension fatigue of 0.5 mm POF under 5 fatigue cycles.

In the program of tension fatigue testing, the maximum applied load was 60% of maximum tensile strength and the minimum applied load was 10% of the maximum applied load (the stress ratio was 0.1), as shown in Figure 4.2.

***Flex fatigue testing***. The flex fatigue properties of all POFs were carried out on Flexometer at 20 ˚C and 65% relative humidity. The testing was aimed to evaluate the flex fatigue lifetime of POF based on the number of bending cycles to failure. The weight *m* could be applied to the free end of POFs. 10 samples can be tested at the same time. 50 times were averaged for each type of POFs.

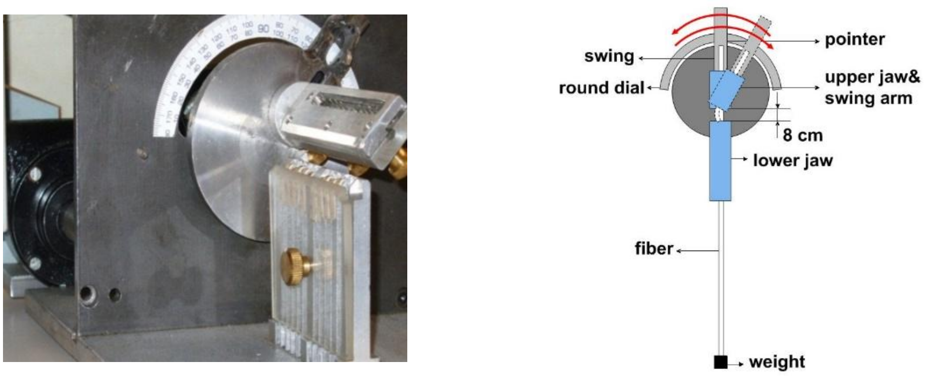


Figure 4.3 Prototype device to measure resistance to bending (left) and corresponding schematic diagram of side view (right).

In this work, the Q-Q plot and Weibull distribution were combined as the exploratory data analysis method to estimate the proper distribution of number of bending cycles *N*. The relations among fiber diameter, number of bending cycles and flexibility were also estimated based on the double logarithmic curves.

# 5 Summary of the results achieved

## 5.1 Tensile properties

The results from tensile testing of all POFs are illustrated in Figure 5.1. It is observed that the fiber diameter has an evident influence on tensile property. Both tensile strength and strain change in the same manner for all POFs, which drop as the fiber diameter rises. There is an opposite relation between tensile strength or strain value and fiber diameter.

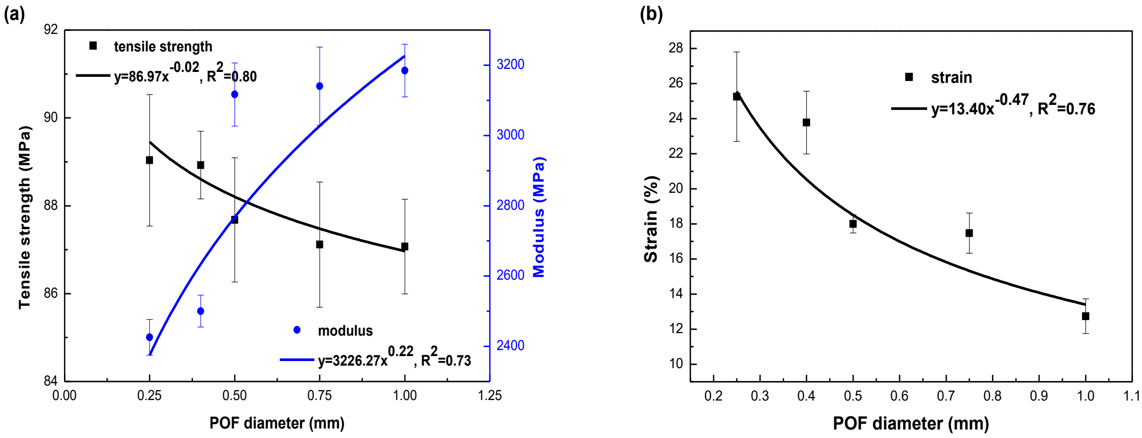


Figure 5.1 POFs with different diameters: (a) tensile strength and modulus; (b) strain.

The phenomenon in tensile strength might be explained by the weakest-link theory. The surface flaws that occur with a statistical nature increase with large surface area, which leads to small tensile strength. The crosshead speed for all POFs keeps constant, that is to say, the extension rate of crosshead for each POF is the same. The thin fiber is generally less stiff than the thick fiber. The ability of deformation for thin fiber is higher and the corresponding extension is larger. Thereby, the thick fiber initiates lower strain rate as expected. Beside, POF has two layers, which are made of different materials, representing different mechanical properties. The thicker the POF diameter, the larger the interface area, the more uneven the adhesion force between layers. Therefore, the thick POF is estimated to fracture with small values of tensile strength and strain.

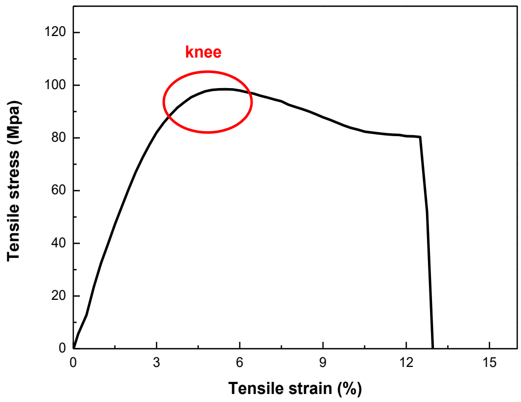


Figure 5.2 Practical stress-strain curve of 0.5 mm POF.

The results of modulus are surprising because it is contrary to the widely accepted assumption that the material modulus is an intrinsic property and should be constant. In present investigation, it points out that the thicker the fiber diameter, the higher the fiber modulus of POF. The similar phenomenon of modulus has been found in [21]. It might reveal that the increases in both strength and strain with small fiber diameter are attributed to the accumulation of each point in fiber, or distributed over the whole mass of fiber. The value of modulus is thus changed as a result of dissimilar increases in strength and strain. The reason for such behavior might be related to the non-linear ductile properties (bi-linear curve with an obvious "knee" shown in Figure 5.2) and the visco-elastic behavior of POF.

## 5.2 Strength distribution

It is observed from Figure 5.3 that it is almost coherent between experimental and theoretical results by using three-parameter Weibull strength distribution when the gauge length *l* is the same as the reference length $l_0$.

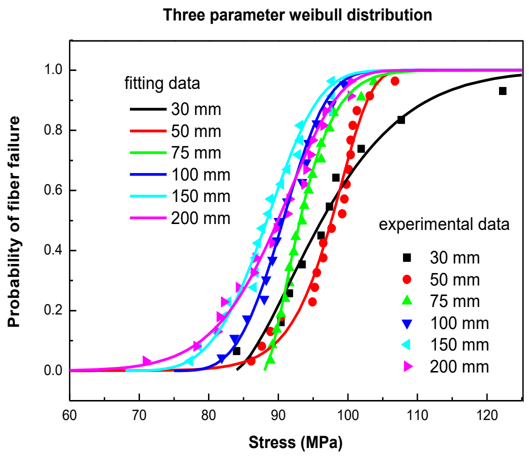


Figure 5.3 Three-parameter Weibull strength distribution of 0.75 mm POF with different gauge lengths ($l = l_0$).

The three-parameter Weibull distribution can be used to predict the dependence of strength on gauge length as well. The Weibull shift parameter is the lower limit of strength, the scale and shape parameters can be obtained from the fitting equation of Weibull distribution based on 1 mm reference length. The relationship between mean fiber strength and gauge length is shown in Figure 5.4.

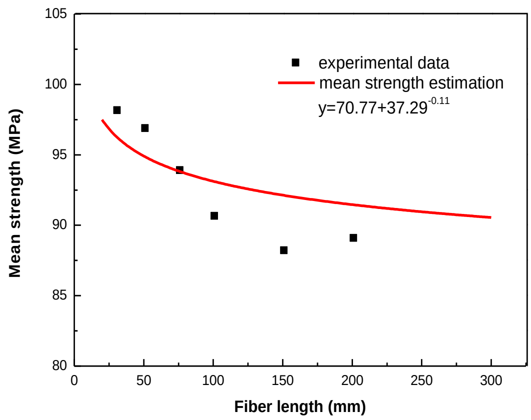


Figure 5.4 Relation between mean fiber strength and gauge length of 0.75 mm POF.

## 5.3 Nanoindentation properties

### *Surface roughness*

In the measurement of topography of samples, the scan size of all images was 5 μm. The POF cladding in Figure 5.5 is on the left-hand side in 2D image, which is in the right part of 3D image. The left part represents the POF core. It is observed that the sample surface is not absolutely smooth, there are some small peaks and valleys in both images, which could be attributed to the manual polishing processes. The value of nanoindentation depth should be larger than the root mean square (RMS) surface roughness in order to minimize the influence of surface roughness on testing results [22]. The value of root mean square surface roughness for each sample is less than 40 nm in this investigation.

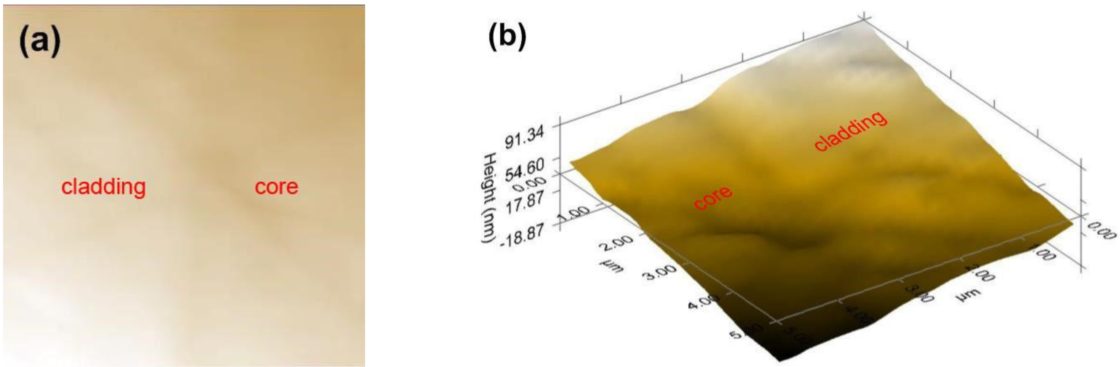


Figure 5.5 Latitudinal cross section of 0.5 mm POF: (a) 2D height image; (b) 3D height image.

***Loading rate effect on nanoindentation creep***

The nanoindentation creep displacements of core and cladding of 0.5 mm POF latitudinal cross section with different loading times are shown in Figure 5.6. First of all, the fitting equation gives a good fit to all experimental data. Secondly, all the curves change in the similar manner. The creep displacement goes up distinctively at the beginning stage of holding period corresponding to the transient creep, and then raises at a relatively gentle increasing rate at the followed steady-state stage [23]. Moreover, the higher the loading rate (the lower the loading time), the bigger the creep displacement. It could be ascribed to the lower strain rate with smaller loading rate, therefore, the longer time is required to reach the maximum load, resulting in the creep deformation during the loading time [24]. It could be also explained by the dislocation substructure that is formed beneath the indenter due to different indentation stresses with various strain rates, and this substructure might play a significant role in the subsequent creep behavior [25].

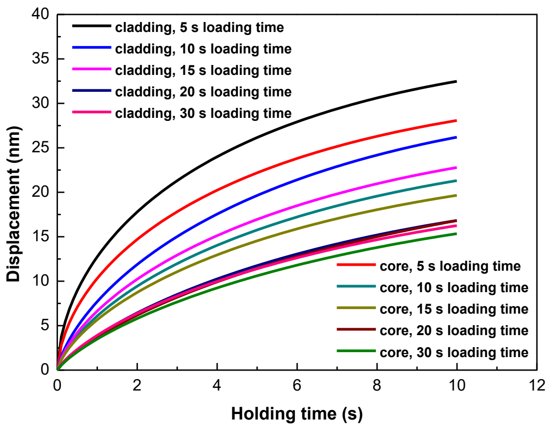


Figure 5.6 Creep displacement of 0.5 mm POF with different loading rates.

Figure 5.7 (a) gives one example with the sample under the condition of 10 s loading time and 10 s holding time, the results show a good fitting match with experimental data based on the coefficient of determination $R^2 = 0.9998$. The corresponding strain rate is shown in Figure 5.7 (a). It is observed that, at the very early beginning of holding period, the displacement increases markedly at a high strain rate from 0.05 ~ 0.025 $s^{-1}$, representing the transient creep. With the increment of holding time, the displacement increases at a gradually saturated strain rate at 0.005 $s^{-1}$ with respect to a steady-state strain [23]. Figure 5.8 (b) shows the related stress exponent.

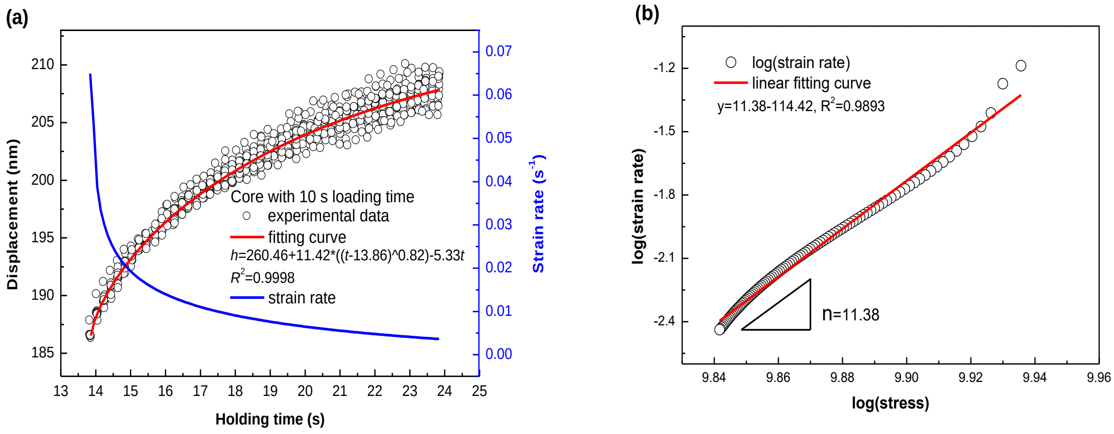


Figure 5.7 0.5 mm POF core with 10 s holding time and 10 s loading time: (a) curves of displacement and strain rate versus holding time; (b) corresponding curve of log(strain rate) versus log(stress).

The variation of stress exponent *n* of 0.5 mm POF at different loading times is displayed in Figure 5.8. It is discovered from this figure that the loading time or loading rate plays a significant role in stress exponent value. The stress exponent decreases gradually with increasing loading time. In this investigation, the maximum indentation depth is approximately 300 nm that is still less than 10% of the sample thickness (1 ~ 1.5 cm), in another word, the influence of substrate is the least [26]. It is convincing that the stress exponent can be calculated from the curve of log(strain rate) versus log(stress), as illustrated in Figure 5.7 (b). It is concluded that the stress exponent is sensitive to the loading time or loading rate, and 0.5 mm POF has a very strong loading rate sensitivity (LRS) in stress exponent.

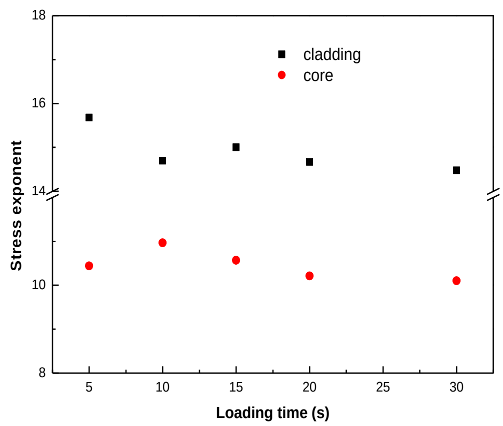


Figure 5.8 Dependence of stress exponent on loading time of 0.5 mm POF.

To understand the creep mechanism of 0.5 mm POF in depth, both values of hardness *H* and elastic modulus $E_r$ are investigated at various loading times, as shown in Figure 5.9. For all samples, both hardness and elastic modulus increase with decreasing loading time or increasing loading rate. When the loading time is lower, the loading rate is higher, the shorter time is allowed to creep, and the smaller indent is created in the end of holding period, which leads to greater hardness due to the smaller contact area. The higher elastic modulus at higher loading rate implies that, with higher loading rate, the less creep occurs during the loading period, and the creep phenomenon could remain in the subsequent unloading time when the elastic modulus is measured.

Based on the theory of Oliver and Pharr method, the contact between indenter tip and sample surface is assumed to be purely elastic during the unloading process. In fact, the contact is far from purely elastic. The creep phenomenon during unloading period could result in the overestimated value of contact stiffness [27-29].

The creep phenomenon in 0.5 mm POF might be induced by plasticity or micro-structure. Given that the fiber is constituted by core and cladding, the interphase between two parts is not clear. The variation of micro-structure in each part may influence its creep phenomenon unexpectedly. It is urgent to figure out the interphase property of 0.5 mm POF. Furthermore, the intrinsic creep mechanism of POF requires more investigation in future.

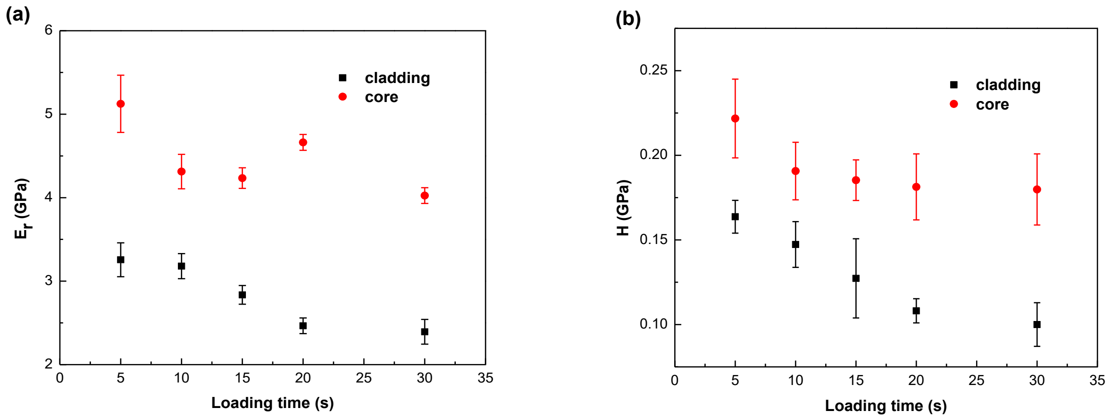


Figure 5.9 0.5 mm POF with 10 s holding time: (a) elastic modulus; (b) hardness.

***Interphase properties***

When the nanoindentation depth declines to 40 nm (close to the value of RMS), the spacing between each two adjacent indentations is around 700 nm, two indents in the transition zone could be observed in both values of hardness and elastic modulus, as shown in Figure 5.10 (a). It is estimated that the interphase width could be in the range of 700 ~ 1900 nm, which is still a wide range and not satisfied enough with the minimum nanoindentation depth.

When the nanoindentation depth is 40 nm, the nanoindentation width is calculated as 302 nm, which means, the minimum safe spacing to avoid the overlapping of the plastic zones between each two adjacent indents is 608 nm. If the spacing is 302 nm, each two adjacent indents would be connected rather than overlapped, while the plastic zones would be definitely overlapped. In this case, the overlapped plastic zones would affect the following results, whereas it is still meaningful to estimate relatively effective interphase width with minimum nanoindentation depth and more sensitive spacing.

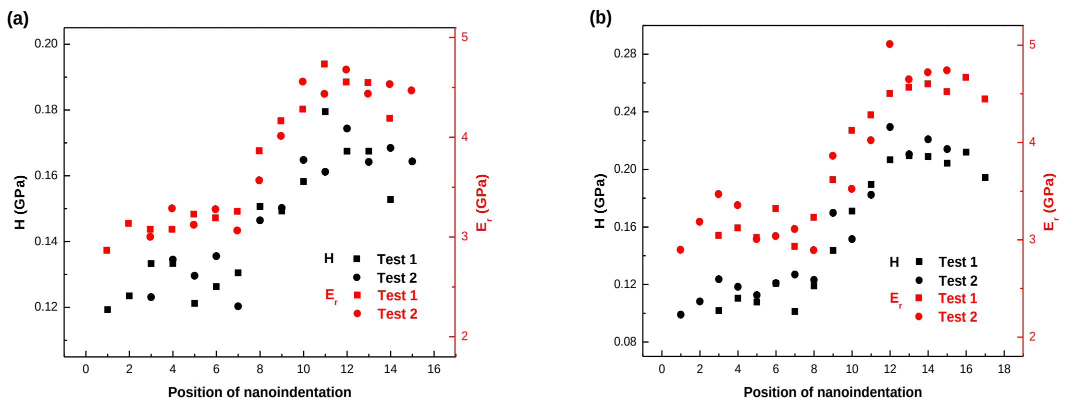


Figure 5.10 Hardness and modulus of 0.5 mm POF with: (a) 40 nm depth and 700 nm spacing; (b) 40 nm depth and 400 nm spacing.

There are three indents during the transition zone and two possibilities of indent locations in the interphase region, as shown in Figure 5.10 (b). Based on the above results, the interphase width could be estimated as 800 ~ 1600 nm.

## 5.4 Tension fatigue properties

***Extension response under constant stress amplitude***

Figure 5.11 depicts the variations of total extension and cyclic extension of selected POFs during tension fatigue testing. Under the same alternating external stress, both total extension and cyclic extension go up evidently with the increment of total fatigue cycles. Moreover, both corresponding increasing rates decline gradually with increasing fatigue cycles.

During the dynamic fatigue testing, the applied strength is generally less than the yield strength. The fundamental reason resulting in the fatigue of plastic material is caused by the visco-elasticity. Under the alternating external stress, the deformation of molecular chains in plastic material always lags behind the stress, which could produce the internal friction, leading to a large amount of heat. This part of heat might accumulate due to the poor thermal conductivity, consequently, the temperature of material itself increases, resulting in the partial softening and melting. The fatigue failure is affected by a lot of factors, such as the internal defects, internal shrinkage, surface scratches, nicks, and roughness of plastic material.

If the unloading time is not sufficient for elastic deformation to recover completely or a part of deformation is resulted from the viscous deformation, the extension at the point of maximum applied load for each cycle would accumulate gradually and the total extension thus goes up with increasing fatigue cycles, as shown in Figure 5.12.

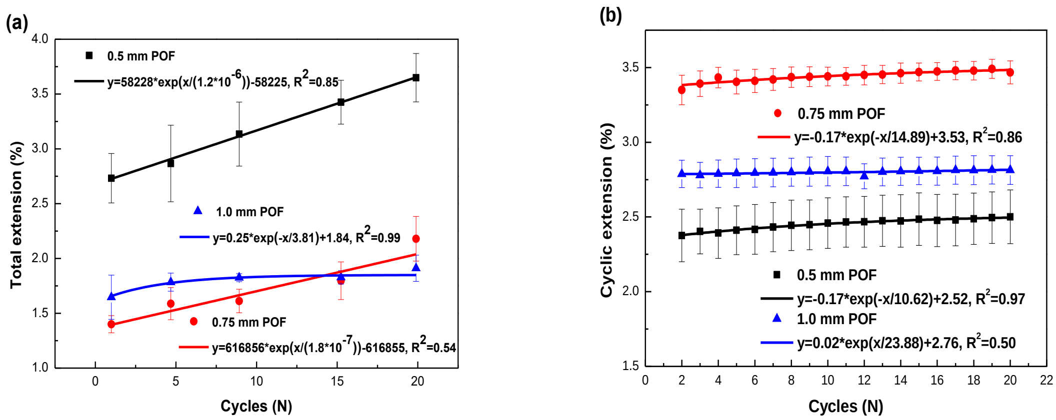


Figure 5.11 POFs in tension fatigue testing: (a) total extension; (b) cyclic extension.

Moreover, it is visible that the fiber diameter has different influences on values of total extension and cyclic extension. POF with 0.5 mm diameter displays higher total extension value and lower cyclic extension value than POFs with 0.75 mm and 1.0 mm diameters. According to the results from tensile testing, the fiber diameter also plays a role in strain value. However, the reason behind it is more complex. The initial gauge lengths and the loading times for three POFs are the same, while the maximum applied loads are different, that is to say, the crosshead speeds are different. The thinner the fiber diameter, the lower the breaking load in tensile testing, the slower the crosshead speed in tension fatigue testing, and the lower the extension rate of fiber. Relatively speaking, the thinner POF has more time to deform during tension fatigue testing. In this case, it is more possible for molecular chains to go straight and slip. Therefore, the total extension should be higher for thinner POF.

The reason for low cyclic extension for 0.5 mm POF could be due to the high value of extension at the point of minimum applied load in each fatigue cycle shown in Figure 5.12. The recovered elastic deformation is the difference between the extension at the point of maximum applied load and the extension at the point of minimum applied load during the same loading cycle, which is considered as the cyclic extension. Given that the unloading time is relatively sufficient for elastic deformation of thinner fiber to recover, it is assumed that a large proportion of viscous deformation might exist during each fatigue cycle and accumulate gradually in thinner fiber.

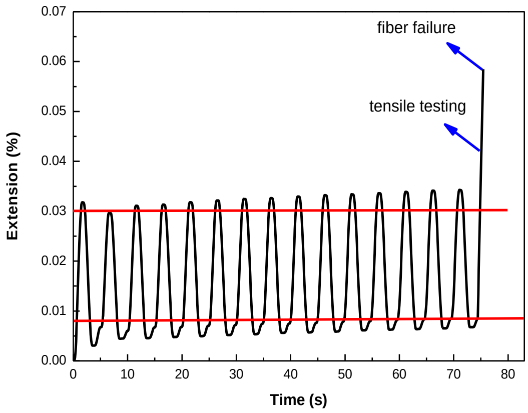


Figure 5.12 Extension versus time curve of 0.5 mm POF in tension fatigue testing with 15 cycles.

***Tensile property after tension fatigue testing***

Figure 5.13 exhibit the changes of strain value of POFs after tension fatigue testing before fiber fracture. The number of fatigue cycles has an evident effect on the tensile properties. With the increase in fatigue cycles, the values of strain decrease markedly with 1 and 5 fatigue cycles, and decline slightly after 5 fatigue cycles. It is observed that the decreasing rates of all values vary with various fiber diameters. The thicker the POF, the higher the decreasing rate. It indicates that the thicker fiber is more sensitive to tension fatigue cycles during dynamic tension fatigue testing. It might be explained by the large interphase area between core and cladding in thick POF, which could result in the high possibility of uniformity problem of interphase adhesion. Another reason could be attributed to the various proportion of core or cladding in all POFs, the respective contributions of core and cladding to the mechanical properties of the whole fiber are not the same in each POF.

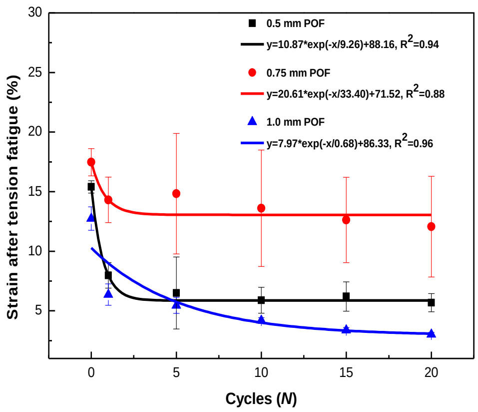


Figure 5.13 Strain value of POFs after tension fatigue testing.

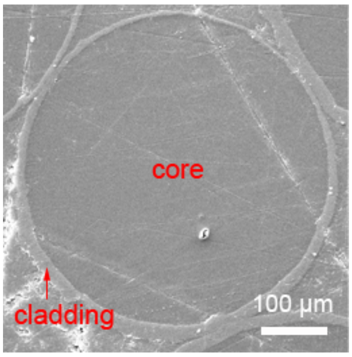


Figure 5.14 SEM image of latitudinal cross section of 0.25 mm POF.

In addition, the cladding is not perfectly even in thickness, as shown in Figure 5.14. Even though the cladding is a relatively thin layer in POF, while its role in fiber properties could not be ignored due to its different material composition.

The material of cladding is composed of PMMA and PTFE. The mixture of PTFE into PMMA can change the properties of material. PTFE is a kind of soft polymers, its modulus is only 0.5 GPa but its strain reaches to 250 ~ 350 % [29]. The different ratio between core and cladding in POFs could lead to the unexpected results of mechanical properties. In a word, it is not easy to investigate the tension fatigue properties of POF with respect to the effect of fiber diameter on extension response.

Moreover, the strain value decreases markedly to a balance state than the values of tensile strength and modulus for all POFs. The strain value is higher than metals but lower than a lot of polymers. POFs applied in this work are relatively brittle, as proved by the fracture images in SEM pictures in Figure 5.15, it is observed that 1.0 mm POF with comparatively smooth and flat fracture surfaces suffers less degradation in tensile properties and fails with less relaxation of damage progression. It implies that POFs present a high strain sensitivity in tensile testing after dynamic tension fatigue testing.

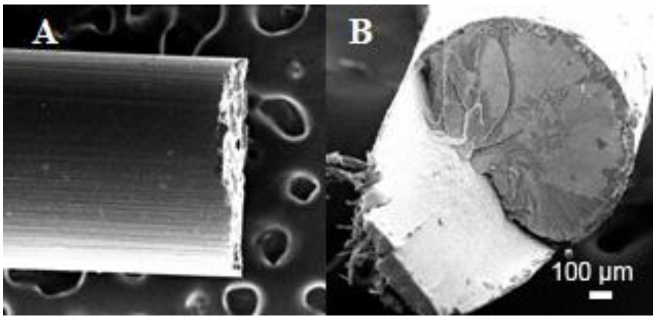


Figure 5.15 Fracture surfaces of 1.0 mm POF from tensile testing: (A) side view; (B) end view.

## 5.5 Flex fatigue properties

***Bending resistance***

Figures 5.16-5.17 illustrate the results from one sample in regard to the Weibull Q-Q plots and Weibull probability plots. It is visible that the combination of Q-Q plot and Weibull distribution can give a good fit to most of experimental data. It indicates from Figure 5.18 that there is the positive relationship between number of bending cycles to fiber failure and flexibility of POF.

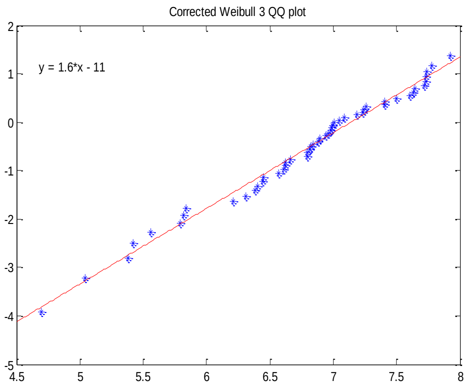


Figure 5.16 Weibull 3 Q-Q plot of 0.25 mm POF.

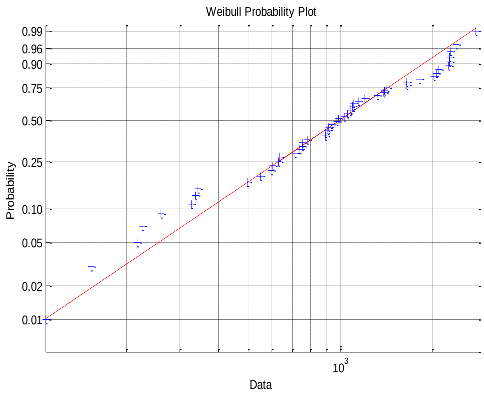


Figure 5.17 Weibull probability plot of 0.25 mm POF.

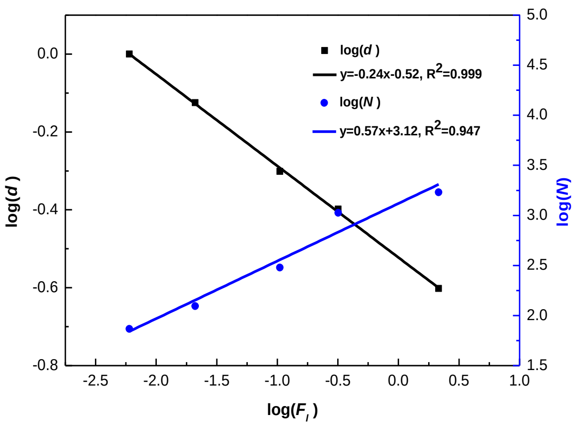


Figure 5.18 Dependences of log(*d*) and log(*N*) on log($F_l$).

***Flex fatigue behavior***

*Flex fatigue life curve*

It is found that the ratio of pretension to ultimate tensile strength $a_c$ (the percentage of pretension) has a significant effect on bending cycles to break, as shown in Figure 5.19. It is well known that, under certain load, the molecular chains are firstly orientated and rearranged; during this period the fiber is stretched straight without any extension. Then the short chains are drawn out from amorphous region. The applied force is undertaken on the long chains until they are broken. Below the value of $a_c$ at 5.55%, 0.25 mm POF might be oriented, resulting in the high flex fatigue resistance to small temporary load. Above this critical value, there is an obvious reduced flex fatigue resistance to larger temporary load. The similar phenomena are found with the value of 88.89%. Especially when $a_c$ is 93.33%, the bending cycle is only 2.36 with relatively weak flex fatigue resistance.

Usually, the ratio of elaborated fatigue strength to ultimate tensile strength for textile materials varies from 50% to 98%. While in this work, the ratio range is boarder. One major reason is due to the POF properties. On the other hand, the testing conditions especially the bending angle or bending speed might affect the results as well. The POFs produced for efficient data transmission generally have the limitation of flexibility, and the bending radius is only eight times of fiber diameter. The large bending angle and fast bending speed could initiate easy destruction of POF due to the limited resistance to flex fatigue.

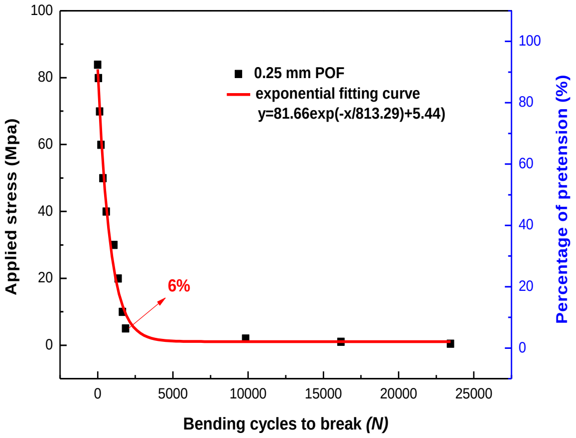


Figure 5.19 *S-N* curve for 0.25 mm POF.

It is observed from Figure 5.20 that there is an obvious plastic deformation on the fracture surface, which is uneven and sloping down from the stretched side (left side) to the compressed side (right side).

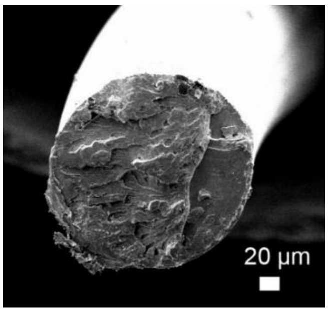


Figure 5.20 Bending fracture of 0.25 mm POF under pretension of 10% of ultimate tensile strength.

*Fatigue sensitivity coefficient*

The values of fatigue sensitivity coefficient (slopes of fitting curves) in this work are less than 1, as shown in Figure 5.21. It could be explained by the high bending angle or bend speed. The number of bending cycles to break is higher with smaller bending angle or bend speed. Therefore, POF is assumed to be more sensitive to flex fatigue with large bending angle or fast bending speed. The core/cladding structure of POF, the variance of core/cladding thickness ratio and the evenness of cladding could also influence the experimental results unexpectedly. In order to understand better, the tensile testing of samples after flex fatigue testing before fiber fracture was also investigated.

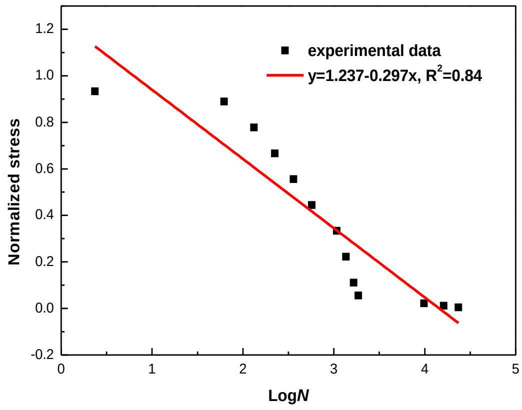


Figure 5.21 Normalized *S-N* curve for 0.25 mm POF.

*Stiffness after flex fatigue testing*

Some samples were taken out from the Flexometer device during the flex fatigue experiments without fiber fracture, in order to investigate the stiffness of 0.25 mm POF. It is evident from Figure 5.22 that, when the pretension is below the upper limit of transition zone which is around 22.22% of ultimate tensile strength, these is no significant modulus degradation with the increase in bending cycles from 10 up to 1000. However, the modulus after flex fatigue testing with 10 bending cycles is less than 5% of the modulus, which means there is an evident loss of modulus during 10 bending cycles. It implies that 0.25 mm POF is very sensitive to flex fatigue.

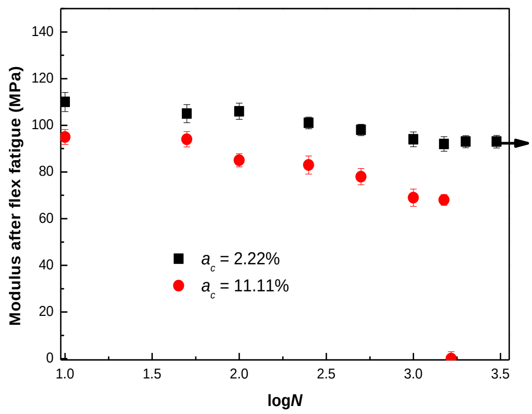


Figure 5.22 Modulus of 0.25 mm POF after flex fatigue testing.

This phenomenon is quite distinct from that of hemp fiber or glass fiber reinforced composites [30]. The bending conditions (bending angle and bending speed) might have an unpredicted influence on POF stiffness, or have a greater impact on POF stiffness than bending cycles in present work. The bending angle and bending speed should be taken into account and studied further.

# 6 Evaluation of results and new findings

## 6.1 Conclusions

It is summarized from the tensile testing that the widely accepted concept about the inverse relation between diameter and tensile strength of single fiber, the weakest-link theory, also conforms to the results of POFs. The strain value of POF decreases with increase in fiber diameter due to the same crosshead speed and high deformation ability of thin fiber. The modulus rises as the fiber diameter goes up, which is not constant as an intrinsic property. It is attributed to the calculation method of modulus dependent on the dissimilar changes of strength and strain.

From the investigation of strength distribution of POF, it is concluded that three-parameter Weibull distribution could be a good model not only for investigation of statistical distribution of fiber strength, but also for estimation of the relation between mean fiber strength and gauge length.

The application of nanoindentation technique in the investigation of local mechanical properties of POF core and POF cladding indicates that there is a positive relation between loading rate and nanoindentation creep deformation. It is observed that POF cladding is softer than POF core. The small nanoindentation depth is expected to give a relatively effective interphase width and the interphase width is estimated to be in the range of 800 ~ 1600 nm roughly, which is still a wide range. To obtain effective interphase properties, the method to create finer surface should be developed. Other techniques such as nanoscrach testing might be also employed to figure out the local mechanical properties and interphase properties of POF [31, 32].

The durability of POF is investigated based on tension fatigue testing and flex fatigue testing. The results from tension fatigue testing indicate that the values of both total extension and cyclic extension go up with increasing fatigue cycles. With the same external stress amplitude, if the unloading time is not sufficient for elastic deformation to recover completely or the viscous deformation occurs, the total extension at each stress peak and the cyclic extension in each loading period would accumulate gradually. In addition, both values are affected by the fiber diameter. It is observed that 0.5 mm POF has higher total extension but lower cyclic extension than thicker POFs. The loading/unloading time for all POFs is the same, while the applied stress is different, therefore, the loading/unloading rate for each POF is various. There is longer time for thin POF to deform, leading to the larger total extension consequently. Due to the presence of probably greater proportion of viscous deformation in the whole deformation, the thinner POF induces lower cyclic value. Furthermore, the tensile testing after tension fatigue testing indicates that POFs present a significant loss in strain value. The thicker the fiber, the larger the loss.

The flex fatigue properties of POF can be characterized with the mean number of bending cycles to break by Flexometer. The combination of Q-Q plot and three-parameter Weibull distribution is effective for estimation of number of bending cycles to break with different POF diameters. There is a positive relation between number of bending cycles to break and flexibility of POF.

The flex fatigue life curve illustrates the decay exponential relation between applied pretension and flex fatigue life time, which are expressed by the percentage of ultimate tensile strength of POF and the number of bending cycles at break, respectively. The flex fatigue resistance of POF increases with decreasing pretension. Meanwhile, the ratio of pretension to ultimate tensile strength of POF varies in a broader range than the common value (50% ~ 90%) of textile materials, and the fatigue sensitivity coefficient of POF is higher than the common value (0.1) of other materials. It is explained mainly by the POF properties and the extensive bending angle or fast bending speed. There is an evident degradation in modulus after flex fatigue testing even though the pretension is below the transition zone of fatigue life curve. However, the modulus decreases slightly after 10 bending cycles.

## 6.2 Other findings

The side illumination of luminous fabrics is important and could be accomplished usually by macro bends of POF in structure design and the surface modifications of POF. Another three methods are introduced here to enhance the side illumination of POF fabrics.

***Laser treatment and titanium dioxide powder***

The side illumination intensity in certain POF distance could be improved by using Titanium dioxide ($TiO_2$) powder and laser treatment. $TiO_2$ is one most widely studied photocatalyst due to high photo-activity, low toxicity, good chemical and thermal stabilities, and cheap price. The refractive index of $TiO_2$ is 2.5, relatively higher than that of POFs, leading to more possibility for light emitting out. Additionally, POF is sensitive to heat. It is believed that the addition of $TiO_2$ particles in fiber cladding would alleviate the heat damage during laser treatment.

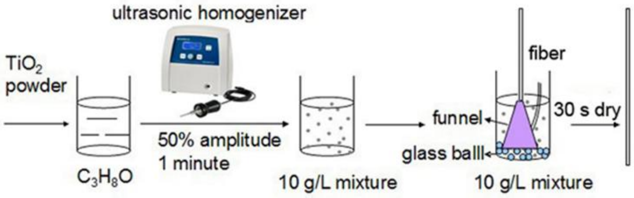


Figure 6.1 Surface modification of POF by $TiO_2$ particles.

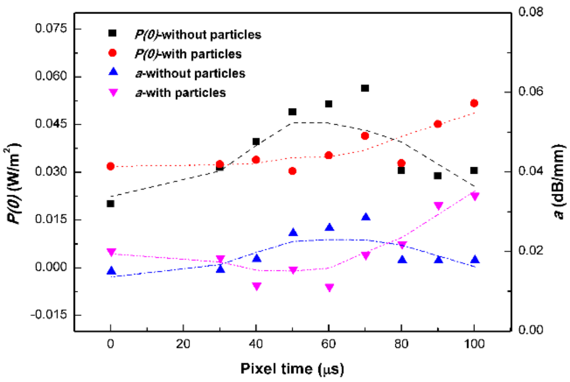


Figure 6.2 Dependence of input illumination intensity and attenuation coefficient on pixel time: dots-experimental data, dash line-smoothed data.

The experimental data indicate that the use of $TiO_2$ particles improves the thermal stability of POF and reduces the transparency of POF simultaneously. If the influence of improved thermal stability on side illumination is dominant, the side illumination might increase in a long distance; if the influence of reduced transparency on side illumination is major, the side illumination might decrease beyond a short distance. Overall, the combination of $TiO_2$ particles and laser treatment can benefit the side illumination to some extent.

***Fluorescent fabrics***

The side illuminating effect of POF is improved by using the woven fluorescent polyester (PET) fabrics which are wrapped on the surface of naked POF. This idea is based on the emission principle of phosphors. The fluorescent fabric first stores the energy from the light source and then releases slowly. When POF wrapped with fluorescent fabric is connected to the light source continuously, the measured side illumination intensity from the surface of sample increases accordingly, as illustrated in Figure 6.3. Moreover, this method could be also applied to even the light diffusion on the surface of sample.

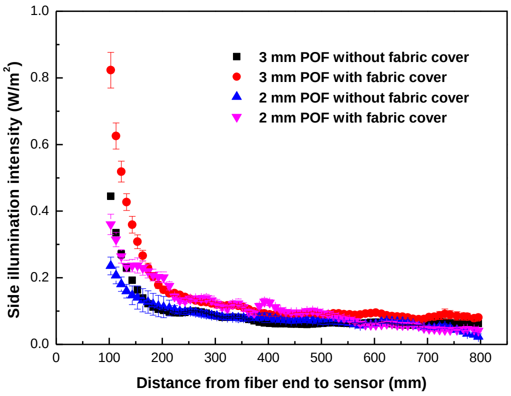


Figure 6.3 Comparison of side illumination intensity of POFs with and without fluorescent PET fabric.

***Lensed POF***

The side illumination intensity of POF could be also enhanced by the lensed end shape, which could be created by the method of $CO_2$ laser cutting. Based on the adjustment of the mark speed of laser treatment and the rotation speed of holding device of POF, different lens shapes could be obtained accordingly. The perfect ball lens in the end of POF could be achieved, as shown in Figure 6.4. The lensed POF can be used as a convex to receive light for light gathering purpose, or applied as a concave to release light for light distribution purpose.

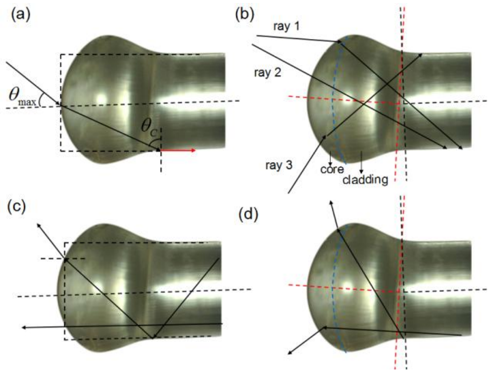


Figure 6.4 Lensed POF: (a-b) light gathering; (c-d) light distribution.

## 6.3 Future work

This thesis work provides the basic knowledge about POF itself, but there are still some confusions left. The future work is introduced as follows.

- Further investigation of the effects of fiber diameter on mechanical properties.
- Study on the fracture mechanism of POF (regarding tensile, bend and torsion) compression by finite element method.
- Improvement of testing methods of side illumination intensity of POF.
- Evaluation of side illumination intensity of POF.
- Simulation of light transmission of side emitting POF.
- Design and fabrication of POF fabrics with dynamic patterns based on above results.
- Investigation of mechanical properties and side illuminating effect of POF fabrics.

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# 8 List of papers published by the author

## 8.1 Publications in journals


1. **J. Huang**, D. Křemenáková, J. Militký, and J. Wiener, *Enhancing Side Illumination of Plastic Optical Fiber by using $TiO_2$ Particles and $CO_2$ Laser*. Journal of Laser Applications, 2015. **27**(3).
2. **J. Huang**, X. Liu, W. B. Li, W. L. Xu, *Preparation and Characterization of Polypropylene/ Superfine Down Powder Blend Films*. Journal of Thermoplastic Composite Materials, 2012, **25**(1): p. 75-88.
3. **J. Huang**, D. Křemenáková, J. Militký, and F. B. Mazari, *Strength Distribution of PMMA Plastic Optical Fiber*. Industria Textila, 2015. **5**: p. 265-268.
4. **J. Huang**, D. Kremenakova, and J. Militky, *Hardness Properties of Plastic Optical Fibers by Nanoindentation.* Tekstil Ve Konfeksiyon, 2014. **24**(4): p. 333-338.
5. **J. Huang**, D. Kremenakova, and J. Militky, *Flex Fatigue Behavior of Plastic Optical Fibers with Low Bending Cycles.* Autex Research Journal, 2015. **15**(2): p. 112-115.
6. **J. Huang**, D. Křemenáková, J. Militký, G. Zhu, and Y. Wang, *Evaluation of Illumination Intensity of Plastic Optical Fibers with $TiO_2$ Particles by Laser Treatment.* Autex Research Journal, 2015. **15**(1): p. 13-18.
7. **J. Huang**, D. Kremenakova, and J. Militky, *Flex Fatigue of Side Emitting Optical Fiber*. Advanced Materials and Engineering Materials, 2013. **683**: p. 425-430.
8. **J. Huang**, D. Křemenáková, J. Militký, V. Lédl, and L. Klara, *Improvement and Evenness of Side illuminating effect of Plastic Optical Fibers by Fluorescent Polyester Fabric*. Textile Research Journal, under review.
9. **J. Huang**, D. Křemenáková, J. Wiener, J. Militký, and M. Kašparová, *Image Analysis of Micro-Lenses Plastic Optical Fiber by Laser Cutting*. Journal of Russian Laser Research, under review.
10. **J. Huang**, D. Křemenáková, J. Wiener, J. Militký, and M. Kašparová, *Enhancement of Illumination Polymeric Optical Fiber with Lens End by $CO_2$ Laser Cutting*. Journal of laser application, under preparation.
11. **J. Huang**, D. Křemenáková, J. Militký, B. Bhera, *Tension-Tension Fatigue of Plastic Optical Fiber*. Fibers and polymers, under preparation.
12. F. B. Mazari, A. Mazari, D. Kremenakova, and **J. Huang**, *Effect of UV-Weathering on Flex Fatigue of Plastic Optical Fiber.* Industria Textila, 2015. **66**(4): p. 171-175.
13. D. Kremenakova, J. Militky, and **J. Huang**, *Characterization of Polypropylene Staple Yarns by Acoustic Dynamic Modulus.* Local Mechanical Properties Ix, 2014. **586**: p. 3-7.


## 8.2 Contribution in book chapters


1. **J. Huang**, D. Křemenáková, J. Militký, and B. Meryová, *Review on Polymeric Optical Fibers*, in *Selected Properties of Functional Materials*, D. Křemenáková, R. Mishra, J. Militký, J. J. Mareš, and J. Šesták, Editors. 2013: Czech Republic. p. 55-92.
2. R. Mishra, J. Militký, V. Baheti, **J. Huang**, B. Kale, M. Venkataraman, V. Bele, V. Arumugam, G. Zhu, and Y. Wang, *The Production, Characterization and Applications of Nanoparticles in the Textile Industry*, in *Textile Progress*. 2014. p. 133-226.


## 8.3 Contribution in conference proceedings


1. **J. Huang** and D. Křemenáková. *Flex Fatigue of Plastic Optical Fiber*. in *Světlanka Workshop*. September 18-20, 2012. Liberec, Czech Republic.

2. **J. Huang**, D. Křemenáková, and J. Militký. *Flex Fatigue Behaviour of Side Emitting Optical Fiber*. in *Structure and Structural Mechanics of Textiles*. December 3-4, 2012. Liberec, Czech Republic.
3. **J. Huang**, D. Křemenáková, and J. Militký. *Loading Time Sensitivity of Nanoindentation Creep of Polymeric Optical Fiber*. in *Structure and Structural Mechanics of Textiles*. December 3-4, 2012. Liberec, Czech Republic.
4. **J. Huang**, D. Křemenáková, and J. Militký. *Flex Fatigue of Side Emitting Optical Fiber*. in *Advanced Materials and Engineering Materials*. December 16-17, 2012. Advanced Materials and Engineering Materials.
5. **J. Huang**, D. Křemenáková, J. Militký, M. Venkataraman, and Y. Wang. *Interphase Properties in Plastic Optical Fiber by Nanoindentation*. in *TexSci*. September 23-25, 2013. Liberec, Czech Republic.
6. **J. Huang**, D. Křemenáková, J. Militký, M. Venkataraman, and G. Zhu. *Mechanical Properties of PMMA Plastic Optical Fibers by Nanoindentation*. in *TexSci*. September 23-25, 2013. Liberec, Czech Republic.
7. **J. Huang** and B. Meryová. *Enhancement of Side emission of Plastic Optical Fibers by Laser treatment*. in *Světlanka workshop*. September 18-20, 2013. Liberec, Czech Republic.
8. **J. Huang**, L. Klara, D. Křemenáková, J. Militký, G. Zhu, and Y. Wang. *Side Illumination of Optical Fibers Wrapped with Fabric*. in *International Conference on Technical Textiles and Nonwovens*. November 6-8, 2014. Indian Institute of Technology Delhi, India.
9. **J. Huang**, D. Křemenáková, and J. Militký. *Nanoindentation Properties of Plastic Optical Fiber Interphase*. in *Nanocon*. November 5-7, 2014. Brno, Czech Republic, EU.
10. **J. Huang**, D. Křemenáková, J. Militký, G. Zhu, and Y. Wang. *Evaluation of Illumination Intensity of Plastic Optical Fibers with $TiO_2$ Particles by Laser Treatment*. in *14th Autex World Textile Conference*. May 26-28, 2014. Bursa, Turkey.
11. **J. Huang**, D. Křemenáková, J. Militký, G. Zhu, Y. Wang, and M. Venkataraman. *Side Illumination of Plastic Optical Fibers Wrapped with Fabrics*. in *Workshop Světlanka*. September 16-19, 2014. Rokytnice nad Jizerou, Czech Republic.
12. **J. Huang**, D. Křemenáková, Y. Wang, G. Zhu, and M. Venkataraman. *Strength Distribution of PMMA Plastic Optical Fiber*. in *Fibers for Progress*. May 21-23, 2014. Liberec, Czech Republic.
13. **J. Huang**, D. Křemenáková, X. Xiong, T. Yang, G. Zhu, and Y. Wang. *Lighting Effect of Plastic Optical Fiber Wrapped with Fluorescent Fabric*. in *20th International Conference Structure and Structural Mechanics of Textiles*. December 1-2, 2014. Liberec, Czech Republic.
14. D. Křemenáková, J. Militký, and **J. Huang**. *Characterization of Polypropylene Staple Yarns by Acoustic Dynamic Modulus*. in *9th Local Mechanical Property*. November 7-9, 2012. Levoča, Slovak Republic.
15. M. Venkataraman, R. Mishra, **J. Huang**, Y. Wang, and G. Zhu. *Evaluation of Thermal Properties of High Performance Nonwoven Padding Fabric*. in *TexSci*. September 23-25, 2013. Liberec, Czech Republic.
16. Y. Wang, J. Wiener, R. Mishra, G. Zhu, M. Venkataraman, **J. Huang**, and J. Militký. *Study on Ozone Treatment of Aramid Fabrics*. in *TexSci*. 2013. Liberec, Czech Republic.
17. Y. Wang, J. Wiener, G. Zhu, and **J. Huang**. *Sorption Property of Polyamide Nanofibrous Membrane on Dyestuff for Purifying Wastewater*. in *Nanocon*. October 16-18, 2013. Brno, Czech Republic.

18. Y. Wang, J. Wiener, G. Zhu, **J. Huang**, and M. Venkataraman. *Apparatus Assembling for Continual Filtration Study of Filter Membrane*. in *TexSci*. September 23-25, 2013. Liberec, Czech Republic.
19. G. Zhu, D. Křemenáková, Y. Wang, **J. Huang**, and J. Militký. *Study on Heat of Liquid Adsorption of Cotton Fabric*. in *Světlanka workshop*. September 18-20, 2013. Liberec, Czech Republic.
20. G. Zhu, D. Křemenáková, Y. Wang, **J. Huang**, M. Venkataraman, and J. Militký. *Evaluation of Thermal Conductivity of Hollow Fiber Padding by Experiment*. in *TexSci*. September 23-25, 2013. Liberec, Czech Republic.
21. G. Zhu, J. Militký, Y. Wang, **J. Huang**, and D. Křemenáková. *Comparison of Effective Thermal Conductivity of Hollow Fibers by Prediction Models and FE Method*. in *AMSED*. September 21-23, 2013. Singapore.
22. J. Militký, D. Křemenáková, **J. Huang**, and A. P. Aneja. *Flex Fatigue of PET/PEN Fibers*. in *4th International Conference on Textile and Material Science* August 27-28, 2014. Ružomberok, Slovak Republic.
23. Y. Wang, J. Winner, G. Zhu, and **J. Huang**. *Constant On-line Apparatus to Investigate Filtration*. in *Fiber for Progress*. May 21-23, 2014. Liberec, Czech Republic.
24. Y. Wang, J. Winner, G. Zhu, and **J. Huang**. *Characterization of Polyamide 6 Nanofibrous Membrane by BET Theory*. in *ICCE-22*. July 13-19, 2014. Malta.
25. Y. Wang, J. Winner, G. Zhu, and **J. Huang**. *Sorption Kinetics Analysis of Acid Dye on Polyamide 6 Nanofibers under Different PH*. in *20th International Conference Structure and Structural Mechanics of Textiles*. December 1-2, 2014. Liberec, Czech Republic.
26. M. Javaid, **J. Huang**, H. Abid, J. Militký, and D. Křemenáková. *Radiation Distribution characterization of Fluorescent Dyed Polyester Fabrics at 633 nm Wavelength*. in *Workshop Světlanka 2015*. September 22-25, 2015. Rokytnice nad Jizerou, Czech Republic.

# Curriculum Vitae

***Personal information***


| | |
|---|---|
| Name | Juan Huang |
| Data of birth | 24/02/1986 |
| Nationality | Chinese |
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***Education***

| | |
|---|---|
| 01/2012-06/2016 | **Ph.D.** in Textile Technology and Engineering, Technical University of Liberec, Czech Republic |
| 09/2008-07/2011 | **M.S.** in Textile Material and Textile Design, Wuhan Textile University, China |
| 09/2004-07/2008 | **B.S.** in Textile Engineering, Wuhan Textile University, China |

***Professional experience***

**Internship experience**

| | |
|---|---|
| 10/2014-12/2014 | Internship for tension fatigue testing in Indian Institute of Technology Delhi, Indian |
| 06/2014-07/2014 | Internship for optical simulation of optical fiber in Toptec Center, Turnov Czech Republic |
| 06/2013-07/2013 | Internship for nanoindentation testing in Wuhan Textile University |

**Project experience**

| | |
|---|---|
| 03/2015-12/2015 | Student Grant Competition (SGS), Czech Republic |
| 03/2014-12/2014 | Student Grant Competition (SGS), Czech Republic |
| 10/2013-12/2013 | Christof project-Surface roughness and waviness of fabrics, Czech Republic |
| 03/2013-12/2013 | Student Grant Competition (SGS), Czech Republic |
| 10/2012-12/2012 | Project of Defence Research and Development Organisation (DRDO), Indian |
| 03/2012-12/2012 | Student Grant Competition (SGS), Czech Republic |

**Conference experience**

| | |
|---|---|
| 03-04/12/2015 | International Material Day in Liberec, Czech Republic |
| 01-02/12/2015 | Advances in Material Engineering in Liberec, Czech Republic |

| | |
|---|---|
| 22-25/09/2015 | Svetlanka Workshop in Rokytnice nad Jizerou, Czech Republic |
| 01-02/12/2014 | Structure and Structural Mechanics of Textiles in Liberec, Czech Republic |
| 06-08/11/2014 | Technical Textiles and Nonwovens in Indian Institute of Technology Delhi, India |
| 05-07/11/2014 | Nanocon in Brno, Czech Republic, EU. |
| 16-19/09/2014 | Svetlanka Workshop in Rokytnice nad Jizerou, Czech Republic |
| 27-28/08/2014 | Textile and Material Science in Ružomberok, Slovak Republic. |
| 26-28/05/2014 | Autex World Textile Conference in Bursa, Turkey |
| 21-23/05/2014 | Fibers for Progress in Liberec, Czech Republic |
| 23-25/09/2013 | TexSci in Liberec, Czech Republic |
| 18-20/09/2013 | Svetlanka Workshop in Liberec, Czech Republic |
| 16-17/12/2012 | Advanced Materials and Engineering Materials in Beijing, China |
| 04/05-07/2013 | Society of Plastics Engineers, Eurotec in Lyon, France |
| 03-04/12/2012 | Structure and Structural Mechanics of Textiles in Liberec, Czech Republic |
| 07-09/11/2012 | Local Mechanical Property in Levoča, Slovakia Republic. |
| 18-20/09/2012 | Svetlanka Workshop in Liberec, Czech Republic |

***Skills***

| | | | |
|---|---|---|---|
| Language (English) | ☆☆☆☆☆ | Cooperation | ☆☆☆☆☆ |
| Office software | ☆☆☆☆☆ | Communication | ☆☆☆☆☆ |
| Originlab | ☆☆☆☆☆ | Organization | ☆☆☆☆☆ |

***Self-evaluation***

I am a friendly and approachable person, can get well along with others. I am active, creative and innovative in academic fields, capable of strong perception and excellent professional skills. Being a teacher or researcher is always my first choice. I hope I could build the bridge between Chinese textile universities and international textile universities or companies.

# Brief description of the current expertise, research and scientific activities

## Doctoral studies

| | |
|---|---|
| Studies | Textile Engineering and Materials Engineering<br>full time |
| Exams | Structure and properties of textile fibers, 05.2012<br>Mathematical statistics and data analysis, 08.2012<br>Textile testing and quality control, 09.2012<br>Optics of solids, 11.2012<br>Experimental technique of the textile, 28.1.2015 |
| SDE | State Doctoral Exam completed on 11.03.2015<br>with the overall result passed. |

## Teaching Activities

| | |
|---|---|
| Teaching | Course of experiments, 02.2013-06.2013 |
| Leading Bachelors/ Master students | No |
| **Research projects** | SGS 2012, project leader, 2012.<br>SGS 2013, project leader, 2013.<br>SGS 2014, project leader, 2014.<br>SGS 2015, project participant, 2015. |
| **Other projects** | DRDO, project participant, 2012. |

# Record of the state doctoral exam

TECHNICKÁ UNIVERZITA V LIBERCI
Fakulta textilní

## ZÁPIS O VYKONÁNÍ STÁTNÍ DOKTORSKÉ ZKOUŠKY (SDZ)

*Jméno a příjmení doktorandky:* **Juan Huang**

*Datum narození:* **24. 2. 1986**

*Doktorský studijní program:* **Textilní inženýrství**

*Studijní obor:* **Textile Technics and Material Engineering**

*Termín konání SDZ:* **15. 4. 2015**

**prospěla** ~~**neprospěla**~~

*Komise pro SDZ:*

| | | *Podpis* |
|---|---|---|
| Předseda: | prof. Ing. Jiří Militký, CSc. | |
| Místopředseda: | prof. Ing. Jaroslav Šesták, DrSc., dr.h.c. | |
| Členové: | doc. RNDr. Jiří Vaníček, CSc. | |
| | doc. Mgr. Irena Lovětinská-Šlamborová, Ph.D. | |
| | Ing. Blanka Tomková, Ph.D. | |

V Liberci dne 15. 4. 2015

*O průběhu SDZ je veden protokol.*

# Recommendation of the supervisor

## Opinion of the dissertation supervisor

## Topic: Selected mechanical properties of polymeric optical fibers

## Author: Juan Huang, MSc.

Flexibility, drapability, durability and intensity of side illumination of POF are critical factors for integration of side emitting polymeric optical fibers (POF) into fabrics, especially for clothing applications. Author of this dissertation work was studying influence of fiber type, fiber diameter, textile structure construction and fabric color (cover of POF is created by special textile containing fluorescent) on selected properties of POF. Results of this work are described in 8 publications in impacted journals, 4 new publications are now under preparation), 2 chapters in books, 23 publications in the conferences (5 abroad, 18 in the international conferences in Czech) and 3 in study workshops in Světlanka (Czech).

The main goal of this work was to study the selected mechanical properties of POF especially elastic modulus and hardness by nanoindentation and other tensile and flex fatigue.
POF history, POF application and main advantages and disadvantages in introduction part were proposed. I have only small comment that now-a-days there are side illumination POF available in the market suitable for range of temperatures from -20°C to 70°C, too.

Part "My state of the art" is based on the general principles of nanoindentation, tensile and flex fatigue of polymers. Many measurements were done by author describing influence of POF diameter, construction and color on the illumination intensity, but it is not included in this part, only small description of influence of polyester fabric with and without fluorescent on the illumination intensity is summarized in chapter "Other findings".

Nanoindentation testing as new approach to study mechanical properties of POF was used. Latitudinal and longitudinal cross-sections of POF in this part including nanoindentation conditions as loading and holding time were studied. Nanoindentation properties, i.e. surface roughness, creep, elastic modulus and hardness were evaluated. Influence of fiber diameter on the elastic modulus and hardness was described, too. Description of interface properties between core and cladding of POF was included.

It was confirmed that fiber diameter has main influence on the tensile and flex properties of POF. It was found that both two and three parameters Weibull distribution for description of strength distribution can be used. Effect of gauge lengths on the strength distribution was evaluated, too.

It was necessary to study tensile and flex fatigue properties, therefore POF with textile cover is produced in single operation on the loom. Tension fatigue properties under constant stress amplitude and tensile properties after tension fatigue testing were described, too.

POF bending resistance play main role for embedding of POF to clothing. For measurement of fibers flex fatigue the special Flexometer device was constructed. Basic result was number of repeated bending cycles of fibers till break. Experimental results by Weibull distribution were described and based on this the lowest possible number of repeated cycles to break was calculated. Good correlation between number of repeated cycles to break and flexibility based on POF diameter and modulus was found. Flex fatigue live curves were found, too.

One very interesting result of this work for new application of POF was in chapt. "Other finding" proposed. One end of POF was created with convex shape by $CO_2$ laser cutting. One part of light rays is through POF cladding and second part is going to fiber end and can be used as other light element.

In conclusion I can say that in this dissertation work nonstandard ways of description of mechanical properties of POF were used. Results of this work are necessary for embedding of optical fibers to clothing and technical textiles especially from point of view flexibility and durability of POF.

.

**The dissertation work meets the specified objectives and I am recommending it for positive defense realization.**

30th April 2016

Assoc. prof. Dana Kremenakova, PhD.
Dept. of Material Engineering
Faculty of Textile Engineering
Technical University of Liberec

# Opponents' reviews

## Opponent's Report of the Dissertation

The author of the dissertation: Juan Huang, M.Eng.
Title: Selected Mechanical Prperties of Polymeric Optical Fiber (POF)
Supervisor: Doc. Dr. Ing. Dana Křemenáková
Dissertation opponent: Doc. Ing. Rydlo Pavel, Ph.D.
Study program: P3106-Textile Engineering
Field of study: 3106V015- Textile Technics and Materials Engineering
Scope of work: 85 pages of text, seven pages of annexes

**Characteristics of the Work:**

The dissertation is focused on the exploration of selected mechanical properties of polymer optical fibres (POF) with the aim to assess the possibilities of application in textile end-uses. Polymer optical fibres (POF) are currently used in many applications. The data communication is an important field of POF applications.

An interesting application, which is the subject of this dissertation, is the use of side-emitting optical fibres with 2-6 mm diameter in textile applications. This allows e.g. the visualization of the contours of people, animals, objects, defining obstacles (stairs, carpet edges) etc.

Chapter 3 describes the basic characteristics of POF, basic relations describing the light propagation in optical fibres and the manufacture of these fibres as well. There is also indicated the construction of POF and the chemical composition of the material used.

In Chapter 4 there are described the methods used for the testing of POF. In this case the tensile test was a basic mechanical characteristic. For this test the tensile testing device Instron was used. Using this test the tensile properties like strength, modulus and strain were obtained.

The nanointendance method and the apparatus Hysitron were used for measuring hardness and tensile modulus. From the measured dependencies there were determined hardness and elastic modulus.

In order to measure the bending properties, the apparatus Flexometer was used. The aim of the testing there was to evaluate the lifetime of POF depending on the number of bending cycles (flex-fatigue testing).

Chapter 5 is an important part of the dissertation, wherein the measurement results including a detailed analysis of the measured dependencies are given. There is possible to indicate the following facts from the analysis that was carried out:

For measuring the tensile properties of laterally radiating optical fibres, depending on their diameter, there was found that the relative strength and elongation decreases with increasing fibre diameter.

With increasing the fatigue cycles, the tensile strength and modulus of elasticity decrease considerably. It is remarkable that the descent rate varies with different fibre diameter; the thicker the fibre, the steeper the descent.

When monitoring the fibre hardness depending on the fibre diameter there was found that the hardness values are higher for the fibres of higher diameter.

When monitoring the number of bending cycles to destruction of fibres there was found that it depends on the fibre diameter. The number of cycles to destruction of the fibre with diameter of 0.25 mm is about 5 times higher than that of 0.5 mm and almost 22 times higher than that of 1 mm.

It would be interesting to verify the above mentioned dependencies using simulation models. The models could be designed, for example, based on the finite element method.

**The Means and Methods used in the Dissertation:**

For the measurements there were used measuring devices that were available at Faculty of Textile Engineering of TUL. In order to characterize local mechanical properties there was used a modern nanoindentation method. The nanoindentation uses a very sharp diamond tip indenter, which will be impressed with a defined force perpendicular to the surface of the sample. During the measurement there is monitored the dependency of the force on the depth of penetration. The data on hardness, Young's modulus or the viscoelastic properties of the sample can then be obtain from the measured dependencies. The range of forces used for the nanoindentation measurements is in the order of μN to mN. The penetration depth in the sample is in the order of nanometers.

**Comments on the Work:**

In this work, the author assumes the POF material to be isotropic. Work does not contain detailed confirmation of this assumption.

POF has a core/shell structure. It would be interesting to create a mathematical model (e.g. Finite Element Method) for this composite material and to verify this model based on the measurements that were carried out by the author.

The graphs in Figures 5.34–5.42 are missing the names of individual axes.

**Benefits of the Dissertation:**

1) Comprehensive view on the mechanical properties of POF with regard to possible applications in textile products;
2) Development of testing methodology;
3) Measurements and analysis of measured results.

**Publications of the Author:**

The author of the dissertation is a co-author of 12 published works in journals and 25 publications at conferences. Furthermore, she participated in selected chapters in two monographs. I can say that her publishing activities are sufficient.

**Questions submitted to the Dissertation:**

1) Has POF stress in torsion been examined? Is knowledge of this stress important for optical fibres integrated in textile materials?

2) Has it been analysed if there is possible to use Hooke's law in general form and to describe the properties of the fibre by stress and strain tensors?

3) The fundamental problem of POF use in textiles there is their energy demand given by the large attenuation of light transmission in comparison with glass fibres. What are the possibilities of increasing the intensity of light emission?

4) POF are resistant to bending, which makes their use in textile applications more difficult. Where do you see the possibilities to improve this deficiency?

**Conclusions:**

In the framework of the dissertation, a methodology of measurement has been developed, numerous measurements were carried out and the obtained data was evaluated and suitably applied to the problem under solving.

Finally, it can be stated that targets of the dissertation have been met.

Based on the above facts **I recommend** This Dissertation for the defense.

V Liberci 21. 3. 2016

Doc. Ing. Pavel Rydlo, Ph.D.
Fakulta mechatroniky,
informatiky a mezioborových studií

Prof. ***Jaroslav Šesták***, Dr.h.c.
*Emeritus Scientist, the Czech Academy of Sciences in Prague*
*Program Auspice, West Bohemian University, Institute of Interdisciplinary Studies*
*Visiting professor, New York State University, Business School in Prague*

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In Prague on April 10, 2016

Subject: Opponent opinion of thesis Juan Huang MSc. on the topic

## Selected mechanical properties of polymeric optical fibers

The thesis contains 85 pages of text, 149 citations and 39 of her own publications, of which are 12 publications in journals and 2 chapters in books. The work is written clearly and shows authors good insight about problems in preparation, properties, testing and illumination behavior of side emitting optical polymer fibers.

Dissertation is written very clearly and especially for me have personal character, as I have in the past personally participated in experimental studies of the suitability of glass materials for optical waveguides.

Review and experimental parts contain:

The history of polymeric optical fibers
Description of reflection and composition of materials
Experimental methods of study
Discussion of results with respect to strength characteristics
Conclusion and Outlook

The main core of dissertation work is study of tensile properties (relationship between fiber strength and gauge length), nanoindentation testing (in terms of hardness property, creep deformation and interphase properties between core and cladding), tension fatigue testing and flex fatigue testing (based on the number of bending cycles to failure and correlation with flexibility).

In dissertation work study of influence of fabric cover with and without fluorescent on the illumination intensity and enhancement of illumination intensity by creation of lensed end fiber shape is also noted.

Although she is the co-author in a chapter in the book "Selected Properties of Functional Materials" (p. 96, cit. 8.2.1.), surely it would be fair to mention also the introductory chapter "Background of fiber optics" (TUL FT 2013), which details outlines history and functionality of optical fibers (in the dissertation chapter. 1st). There also no mention of a recent book TUL "Recent Developments in fibrous material science" (TUL FT 2015).

The work could be given a deeper discussion on the definition of the author's own opinion on optimizing processes, including estimates of the prospects for future development.

Overall, I am satisfied with the work, processing and content. I evaluate and classified this work into the standard dissertations handed to the related fields of material research.

The work meets the requirements for the doctoral thesis and meets the requirements of both the Ministry of Education and TUL - Technical University in Liberec and therefore

**I recommend**

the work for defense and author to award a PhD title.

Sincerely

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